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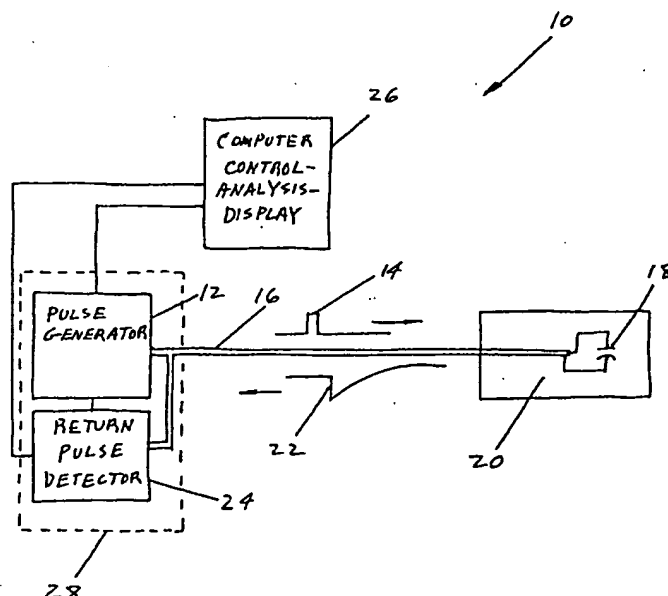
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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/US98/18736 (22) International Filing Date: 9 September 1998 (09.09.98)  (30) Priority Data: 08/926,731 10 September 1997 (10.09.97) US  (71) Applicant: MATERIAL SENSING & INSTRUMENTATION, INC. [US/US]; 772 Dorsea Road, Lancaster, PA 17601 (US). (72) Inventor: HAGER, Nathaniel, E., III; 772 Dorsea Road, Lancaster, PA 17601 (US). (74) Agent: HUNTLEY, Donald, W.; Huntley & Associates, 1105 North Market Street, P.O. Box 948, Wilmington, DE 19899-0948 (US).		(81) Designated States: CA, DE, GB, RU, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  Published With international search report.

(54) Title: METHOD AND APPARATUS FOR IN-SITU MEASUREMENT OF POLYMER CURE STATUS



## (57) Abstract

An apparatus and method for determining the cure state of thermosetting polymers using time domain reflectometry. A miniature capacitor (18) is constructed at the end of a controlled transmission line (16) which is immersed in the curing polymer (20) so that the polymer (20) is the dielectric of the capacitor (18), and step function voltage pulses (14) are fed to the transmission line, while the reflected signal (22) from the line (16) is monitored. The amplitude and decay characteristics of the reflected signals (22), which are related to the degree of cure and the viscosity, respectively, are fed to a computer (28) for interpretation and display (26).

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## BACKGROUND OF THE INVENTION

This invention deals generally with electrical measuring and testing, and more specifically with the determination of the non-electric property of the state of cure of a thermosetting polymer by the use of time domain reflectometry.

The degree of cure of thermosetting polymers is of considerable interest in industry because the cure status determines the strength of a part and also whether it can be subjected to subsequent manufacturing processes. However, since the cure status of the internal portion of a part can not be evaluated visually, considerable effort has been expended to find methods to accurately evaluate the cure state.

Several electrical methods to determine cure state have been used. Among them are the measurement of impedance (U.S. Patent 5,432,435 to Strong et al), reflection of continuous microwave energy (U.S. Patent 5,059,914 to Lacombe et al), and dielectric properties (U.S. Patent 4,777,431 to Day et al). Carrozzino et al have also performed laboratory tests (noted in Polymer Engineering And Science. March 1990, Vol. 30, No. 6, p 366) on the use of time domain reflectometry to evaluate the degree of cure. However, Carrozzino concluded that such an approach had poor accuracy and repeatability, and the publication did not disclose the use of in-situ sensors or the measurement of viscosity.

## SUMMARY OF THE INVENTION

The present invention provides a time domain reflectometry apparatus which furnishes highly accurate and repeatable measurements of the viscosity and percent of cure of polymers. The invention combines the simplicity of electrical sensing with miniaturization available from high frequency techniques in a time domain reflectometry measuring device. Such an apparatus is usable for in-situ monitoring of the cure status of composites, and is important for overcoming quality control problems, increasing production speed, and improving the uniformity of composite

1 manufacture, especially in critical structures.

2 In the preferred embodiment of the invention, a sensor embedded within the  
3 part being constructed receives and reflects a fast rise time pulse, and the reflected  
4 transient signal relates to the dipole rotation occurring in the microwave frequency  
5 range for a particular epoxy dipole. Because of this phenomenon, the reflected signal  
6 can be directly related to viscosity and percent of cure. In experimental tests, changes  
7 in the signal have been followed during processing and compared to other test  
8 methods to establish information in regard to the relationship of the reflected signal to  
9 the state of cure and to the viscosity.

10 The sensor is a miniature capacitor constructed at the end of a miniature  
11 transmission line which is immersed in the curing polymer. Step function voltage  
12 pulses are fed to the transmission line, and the reflected signal from the transmission  
13 line is monitored. The amplitude of the reflected pulse signals, which is related to the  
14 degree of cure, and the decay characteristics of the reflected pulse signals, which are  
15 related to the viscosity, are then fed to a computer for interpretation and display.

16 The invention thereby furnishes a real time measurement of the state of cure of  
17 the polymer, and the transmission line and sensor, which remain in place after the  
18 material is cured, can actually be used with the same signal generator and signal  
19 processing system to later check for cracks or discontinuities which might develop in  
20 the cured part at a later time.

#### 21 BRIEF DESCRIPTION OF THE DRAWINGS

22 FIG. 1 is a simplified schematic diagram of the preferred embodiment of the  
23 invention.

24 FIG. 2 is a partial cross section side view of the sensor and transmission line  
25 of the alternate embodiment of the invention.

FIG. 3 is a top view of the sensor and transmission line of an alternate

1 embodiment of the invention.

2 FIG. 4A and 4B are graphs of the typical variations in the reflected pulse with  
3 time as polymers cure.

4 FIG. 5 is a graph showing how the decay of reflected pulses changes with  
5 viscosity.

6 FIG. 6 is a graph showing how the reflected pulses change with reduced dipole  
7 rotation as cure progresses.

#### 8 DETAILED DESCRIPTION OF THE INVENTION

9 FIG. 1 is a simplified schematic diagram of the preferred embodiment of cure  
10 monitoring apparatus 10 of the invention in which pulse generator 12 supplies step  
11 function incident pulse 14 to transmission line 16. Capacitor sensor 18, which is  
12 located at the remote end of transmission line 16, is immersed within curing polymer  
13 20 and reflects return pulse 22 back to pulse detector 24. The timing of pulse  
14 generator 12, the analysis of return pulse 22, and the display of the test parameters and  
15 results are performed by computer 26.

16 The parameters of the reflected pulse which indicate the viscosity and cure  
17 status of polymers are the variations in the decay time for viscosity and variations of  
18 the amplitude for percent of cure. These are affected by the dipole rotation spectrum  
19 of the polymer. Changes in the reflected pulse parameters are actually indications of a  
20 more sophisticated change in frequency characteristics of the polymer, so that  
21 measurements of the changing characteristics of the reflected pulse actually measure  
22 changes in the materials' frequency characteristics, such as changes in loss peak  
23 frequency.

24 The frequency characteristics can also be measured by direct application of a  
25 signal sweeping through a wide spectrum of frequencies and spectrum analysis of the  
returning signals, but such an arrangement, although perhaps easier to interpret, is

1 more difficult to measure. The pulse technique used in the invention uses a fast  
2 risetime pulse, a few nanoseconds or less, which is well understood in the art to  
3 actually include the same wide spectrum of frequencies. The reflected pulse then  
4 actually includes the desired frequency information, and a Fourier Transform can also  
5 be used to arrive at the dipole rotation spectrum and the loss peak frequency.

6 However, the desired information of percent cure and viscosity can also be  
7 secured by making simple measurements on the reflected pulse.

8 The invention's preferred embodiment of a method of measuring cure status,  
9 that is, viscosity and percent of cure of a polymer, is:

10 A) immersing at least one end of a transmission line into a polymer which  
11 is curing, with a capacitor connected to the immersed end of the transmission line, and  
12 the capacitor constructed so that its dielectric is the polymer into which it is  
13 immersed;

14 B) generating one or more step function voltage pulses and feeding the  
15 pulses to the transmission line;

16 C) receiving reflected pulses back from the capacitor at the end of the  
17 transmission line; and

18 D) analyzing changing characteristics of the reflected pulses to establish  
19 the viscosity and status of cure of the polymer in which the transmission line is  
20 immersed.

21 In most installations pulse generator 12 and pulse detector 24 are actually  
22 included in one unit, sampling head 28. In the preferred embodiment, sampling head  
23 28 is a Hewlett Packard model 54750 Time Domain Reflectometry Oscilloscope  
24 which has a 35 ps input transient and a 20 GHz detection bandwidth.

25 In the preferred embodiment, incident pulse 14 is 200 millivolts, with a rise  
time of 35 picoseconds. The pulse length and repetition rate are not critical as long as

1 the pulse length is long enough and the repetition rate is slow enough so that they do  
2 not result in additional pulses being generated during the 1 nanosecond to 10  
3 microseconds after the beginning of the reflected pulse when pulse analysis is being  
4 performed. The desirable pulse risetime and detector sampling resolution is in the  
5 range of 1 picosecond to 10 nanoseconds. Pulses can also be produced in bursts to  
6 produce a lower effective frequency so that data is transferred to computer 26 at a  
7 lower effective rate.

8 Transmission line 16 can be any type of controlled impedance high frequency  
9 transmission line. As illustrated in FIG. 2 and FIG. 3, this includes both coaxial and  
10 flat ribbon, so called "Stripline", transmission lines. For the preferred embodiment, a  
11 .25 mm, 50 ohm, flat ribbon line is used. The length of transmission line 16 was  
12 chosen to avoid  $\frac{1}{4}$  wave resonance, and the line itself must be free of connectors  
13 which add undesirable signal distortions and reflections.

14 Sensor 18, which is immersed in curing polymer 20, is essentially a miniature  
15 capacitor whose fringing electric field extends into the polymer. In the preferred  
16 embodiment, it is a 1 to 2 picofarads capacitance formed near end 30 of transmission  
17 line 16, as shown in FIG. 2, but it is practical to use a capacitance in the range of 1 to  
18 10 picofarads.

19 FIG. 2 is a top view of Sensor 32 and transmission line 34 of the preferred  
20 embodiment of the invention. Sensor 32 and transmission line 34 are constructed as  
21 planar stripline components. Such devices are conventionally fabricated on a thin  
22 insulating substrate, and center conductor 36 is centered between coplanar ground  
23 planes 38. Transmission line 34 of the preferred embodiment of FIG. 2 is constructed  
24 with a 50 ohm impedance along the transmission path, with an interlaced finger  
25 structure 40 at the end of transmission line 34 to form capacitor 42.

Transmission line 34 and capacitor 42 are fabricated on copper laminated



1   teflon sheets of .1 to 1 millimeter thickness, and the pattern is made by  
2   photolithography and chemical etch methods. The material between the fingers 40 is  
3   completely etched away so that it will be replaced by the curing polymer within which  
4   capacitor 42 will be immersed. If a greater capacitance is desirable, the fingers 40 can  
5   easily be made longer, thus increasing the effective surface of the capacitor. In fact,  
6   one way in which the stripline capacitor is constructed is to manufacture the capacitor  
7   section 42 of stripline transmission line 34 much longer than will be needed and to  
8   merely cut it down to secure the desired capacitance in the 1 to 10 picofarad range.

9         In an alternate embodiment shown in FIG. 3, capacitor 18 is formed on  
10   transmission line 16 by first removing a short length of outer coaxial shield 46.  
11   leaving a 2 to 3 mm length of insulating sleeve 48 exposed. The entire surface of  
12   exposed insulating sleeve 48, including end 49, is then covered with electrically  
13   conductive layers 50 and 52, which are shown in cross section. Conductive layers 50  
14   and 52 are typically metal, although they can be other electrically conductive  
15   materials. They can be applied using conventional methods, such as evaporation or  
16   chemical plating. At this stage of construction, conductive layers 50 and 52 are joined  
17   because they have been formed as one continuous layer, and they actually create a  
18   short circuit between center conductor 54 and outer shield 46. However, the thickness  
19   of the conductive layers is not critical since there will be no significant current flow  
20   through them.

21         The final stage of the construction of capacitor 18 is simply forming gap 56  
22   along the length of insulating sleeve 48 and separating conductive layers 50 and 52.  
23   Gap 56 extends through conductive layers 50 and 52 and insulating sleeve 48 to center  
24   conductor 54, and thus destroys the previously formed short circuit. Gap 56 thereby  
25   functions as the dielectric space of a capacitor whose "plates" are conductive layers 50  
   and 52, which are connected to inner conductor 54 and outer shield 46, respectively.

1 Gap 56 is eventually filled by the curing polymer within which capacitor 18 is  
2 immersed. Gap 56 is formed by conventional methods such as laser cutting, or  
3 chemical etch methods. If a greater capacitance is desirable, gap 56 can be formed as  
4 two interlaced finger structures, thus increasing the effective surface of capacitor 18.

5 FIG. 4 A and B are graphical representations of the typical variation in  
6 amplitude of reflected signal 22 (FIG. 1), in millivolts, against cure time, in minutes,  
7 as polymer 20 (FIG. 1) goes through a normal curing process. The readings for FIG.  
8 4A were taken at 1 nanosecond after the start of the reflected pulse, and are therefore  
9 close to the peak of the reflected pulse. The readings for FIG. 4B were taken at 50  
10 nanoseconds after the start of the reflected pulse, and are therefore well into the  
11 decaying "tail" of the pulse. The material used in these tests was Hexcel 8552. The  
12 curve is essentially similar for most thermosetting polymers. The temperature at  
13 which the samples were held during the tests was between 25 - 175 degrees Celsius.  
14 With such data available to computer 26 of FIG. 1, it is a simple task to establish  
15 when a polymer has experienced equivalent signal reductions and therefore to  
16 establish the state of cure by comparing the changing characteristics of the reflected  
17 pulses to characteristics of pulses reflected during previous controlled tests of curing  
18 polymers. This is easily accomplished by the use of computer 26 whose memory can  
19 include the previous test results. Moreover, controlled tests can be run and put into  
20 memory for any new type of material so that later production testing equipment can  
21 evaluate such new materials.

22 However, previous test results are not a necessity. A preferred method of  
23 analysis is to measure and compare the changing amplitudes at two points on the  
24 decaying tail of the pulse for a series of reflected pulses over a period of time. As  
25 indicated in FIG. 4, typically, one of these observation points on the reflected pulses is  
less than 10 nanoseconds after the reflected signals begin, and the other observation

1 point on the reflected pulses is greater than 15 nanoseconds after the reflected pulses  
2 begin. These time ranges are chosen because, as can be seen on FIG. 5, they exclude  
3 the region in which change is minimal for the viscosity indicator factor.

4 FIG. 5 is a graph showing the manner in which the reflected pulses change as  
5 viscosity of the typical polymer changes. As can be seen from the two curves on FIG.  
6 5, as viscosity increases as the polymer cures, the amplitudes of the reflected pulses  
7 are decreased at short times and increased at long times. A simple ratio of the  
8 amplitudes at the short time measurement point and at the long time measurement  
9 point can therefore indicate the change in viscosity of the polymer.

10 It is important to recognize that the curing process is usually preceded by a  
11 period in which the temperature of the polymer is increased, and during this time,  
12 when no curing is taking place but the viscosity is decreasing, the ratio of amplitude  
13 of the signal at short times to the amplitude of the signal at long times is an indicator  
14 that this process is taking place.

15 FIG. 6 is a graph showing the manner in which the reflected pulses change  
16 with reduced dipole rotation as cure of the typical polymer progresses. In this case the  
17 amplitudes of the reflected pulses are reduced at all points. In fact, the completion of  
18 the curing process can be determined when this reduction in amplitude is no longer  
19 occurring, indicating that the cure is complete. It is interesting to note that the  
20 absolute amplitude of reflected pulses at this end point of solidification should always  
21 be approximately the same for the same sensor and the same material. Therefore, in a  
22 production situation, if the amplitude reduction has stopped, but the absolute value of  
23 the amplitude is not within an appropriate range, it is an indication that there is an  
24 undesirable variation in the material.

25 The present invention thereby provides an accurate and reliable means to  
evaluate cure status of various polymers, and is capable of performing the task in

1 either the laboratory or the production environment.

2       It is to be understood that the form of this invention as shown is merely a  
3 preferred embodiment. Various changes may be made in the function and  
4 arrangement of parts; equivalent means may be substituted for those illustrated and  
5 described; and certain features may be used independently from others without  
6 departing from the spirit and scope of the invention as defined in the following claims.

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## I CLAIM:

1. A method of measuring viscosity and percent of cure of a polymer comprising:
  - immersing at least one end of a transmission line into a polymer which is curing, with a capacitor connected to the immersed end of the transmission line, and the capacitor constructed so that its dielectric is the polymer into which it is immersed;
  - generating at least one step function voltage pulse and feeding it to the transmission line;
  - receiving a reflected pulse back from the capacitor at the end of the transmission line; and
  - analyzing the characteristics of the reflected pulse to establish the condition of the polymer in which the transmission line is immersed.
2. A method of Claim 1 wherein analyzing the reflected pulse comprises comparing characteristics of the reflected pulse to characteristics of pulses reflected during previous tests of curing polymers.
3. A method of Claim 1 further including generating a series of pulses and analyzing the reflected pulses by comparing the changing amplitudes of the reflected pulses over time to determine the state of cure of the polymer.
4. A method of Claim 1 further including generating a series of pulses and analyzing the reflected pulses by comparing the changing decay curves of the reflected pulses over time to determine the viscosity of the polymer.
5. A method of Claim 1 further including transforming the reflected pulses into the frequency domain and analyzing the resulting frequency of the dipole rotation spectrum to determine the viscosity of the polymer.
6. A method of Claim 1 further including transforming the reflected

1 pulses into the frequency domain and analyzing the resulting loss peak frequency to  
2 determine the state of cure of the polymer.

3 7. A method of Claim 1 wherein analyzing the reflected pulses comprises  
4 comparing over time the amplitudes of the reflected pulses at a point on the reflected  
5 pulses which is less than 10 nanoseconds after the reflected pulses begin to the  
6 amplitudes of the reflected pulses at a point on the reflected pulses which is greater  
7 than 15 nanoseconds after the reflected pulses begin.

8 8. An apparatus for measuring viscosity and percent of cure of a polymer  
9 comprising: a pulse generator generating at least one step function voltage pulse;  
10 a transmission line connected to and receiving pulses from the pulse generator,  
11 with one end of the transmission line immersed in a polymer which is curing;  
12 a capacitor connected to the immersed end of the transmission line and also  
13 immersed in the polymer which is curing, the capacitor being constructed so that its  
14 dielectric is the polymer into which it is immersed;  
15 a pulse detector connected to the transmission line and receiving a signal from  
16 the transmission line which is a pulse reflected from the immersed capacitor;  
17 and an analyzer means interconnected with the pulse detector, the analyzer  
18 means being capable of measuring the changes in reflected pulses as the polymer  
19 cures.

20 9. An apparatus of Claim 8 wherein the pulse generator and the pulse  
21 detector are included in the same instrument.

22 10. An apparatus of Claim 8 wherein the transmission line is a flat line  
23 with a central conductor, insulating strips on both sides of the central conductor and  
24 outer conductors on the opposite sides of the insulating strips from the central  
25 conductor, and the capacitor is integrated into the transmission line by forming an  
interlaced finger structure adjacent to the end of the transmission line.

1           11.    An apparatus of Claim 10 wherein the central conductor and the outer  
2 conductors adjacent to the end of the transmission line form two interlaced finger  
3 structures so that the central conductor and the outer conductors have an increased  
4 area adjacent to the end of the transmission line.

5           12.    An apparatus of Claim 8 wherein the transmission line is coaxial with  
6 an inner conductor, an insulator, and an outer conductor,  
7           and the capacitor is integrated into the transmission line by forming a gap in  
8 the insulator and outer conductor adjacent to the end of the transmission line, and  
9 connecting the separate part of the outer conductor to the central conductor of the  
10 cable.

11           13.    An apparatus of Claim 8 wherein the capacitance of the capacitor is in  
12 the range of between 1 and 10 picofarads.

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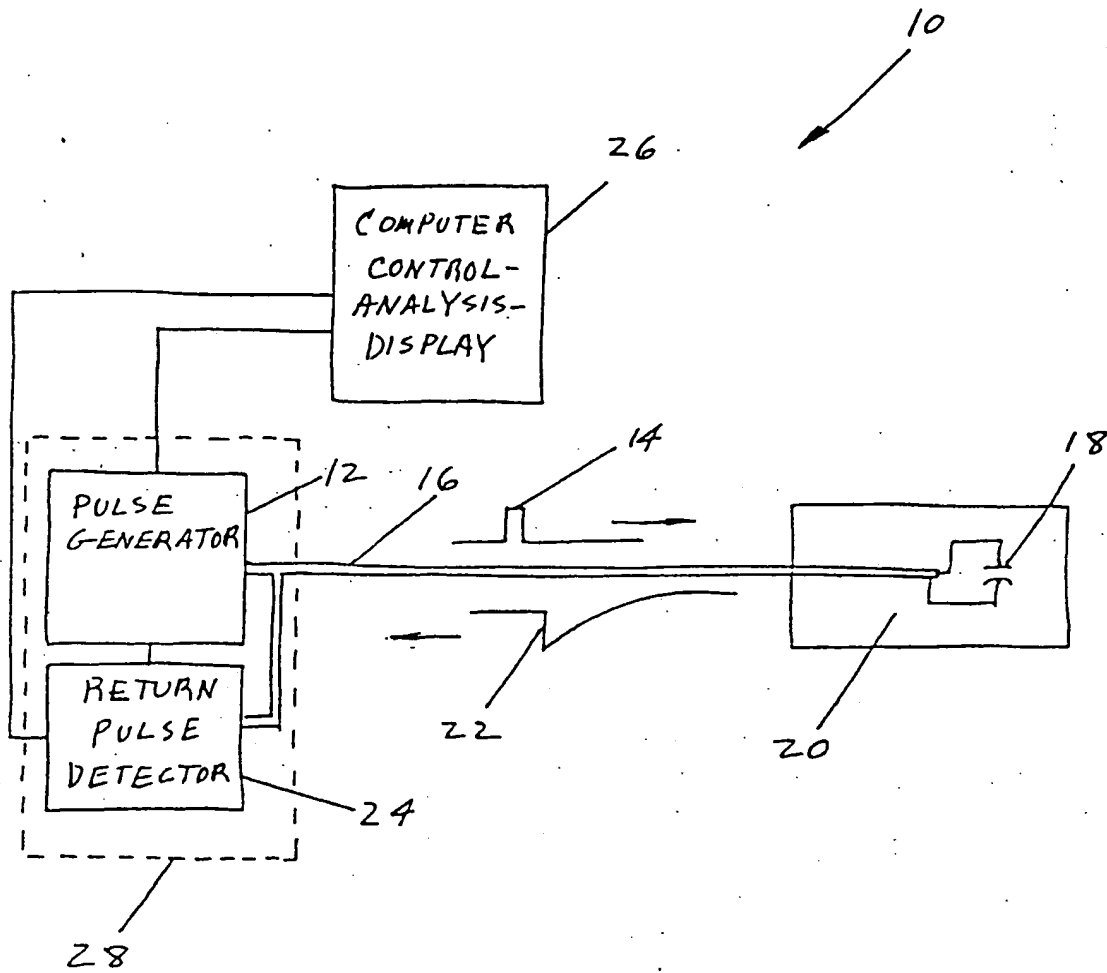


FIG. 1



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Figure 2 – Stripline Sensor

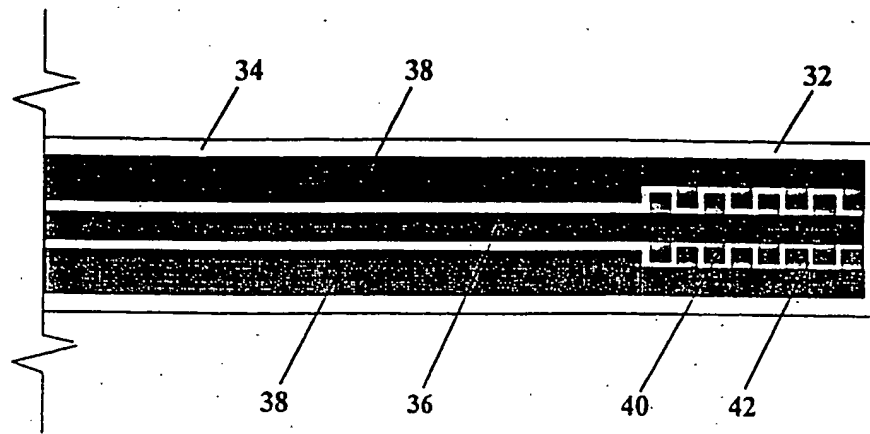
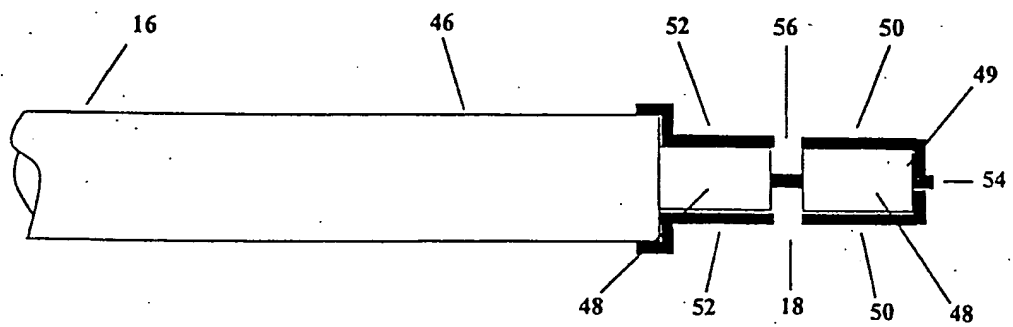
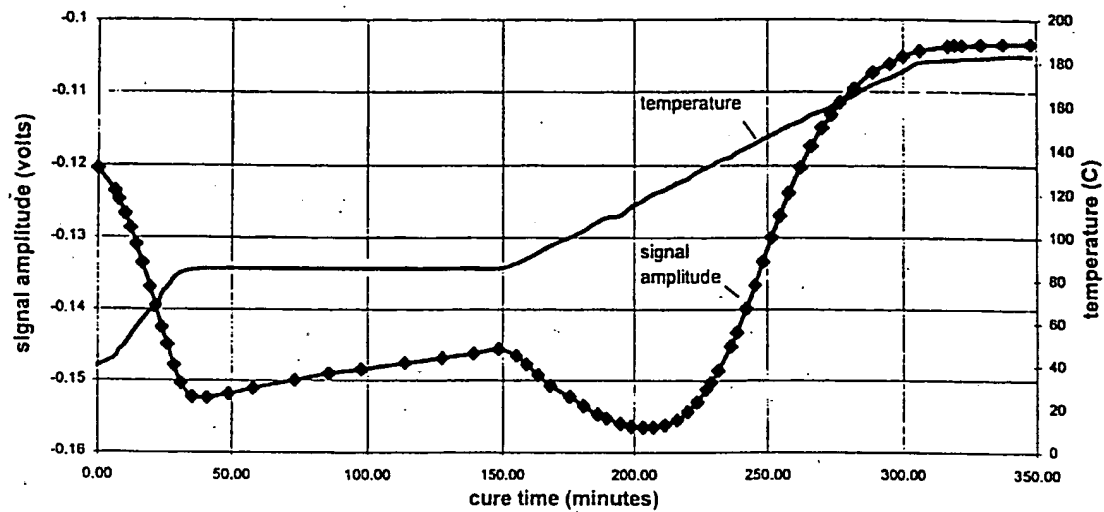
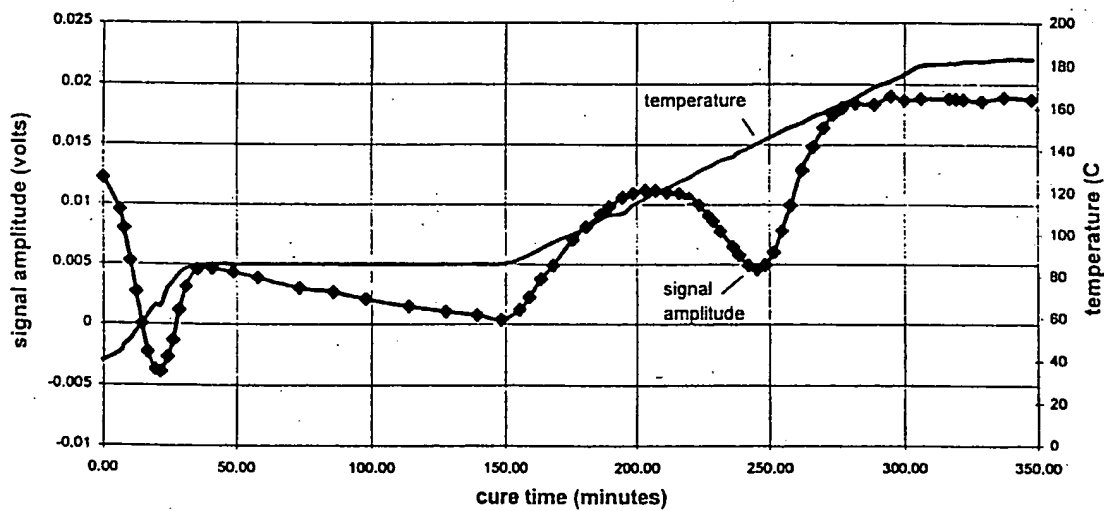


Figure 3 – Coaxial Sensor



**Fig 4a - Reflected Pulse Amplitude at 1.0 Nanoseconds****Fig 4b - Reflected Pulse Amplitude at 50 Nanoseconds**

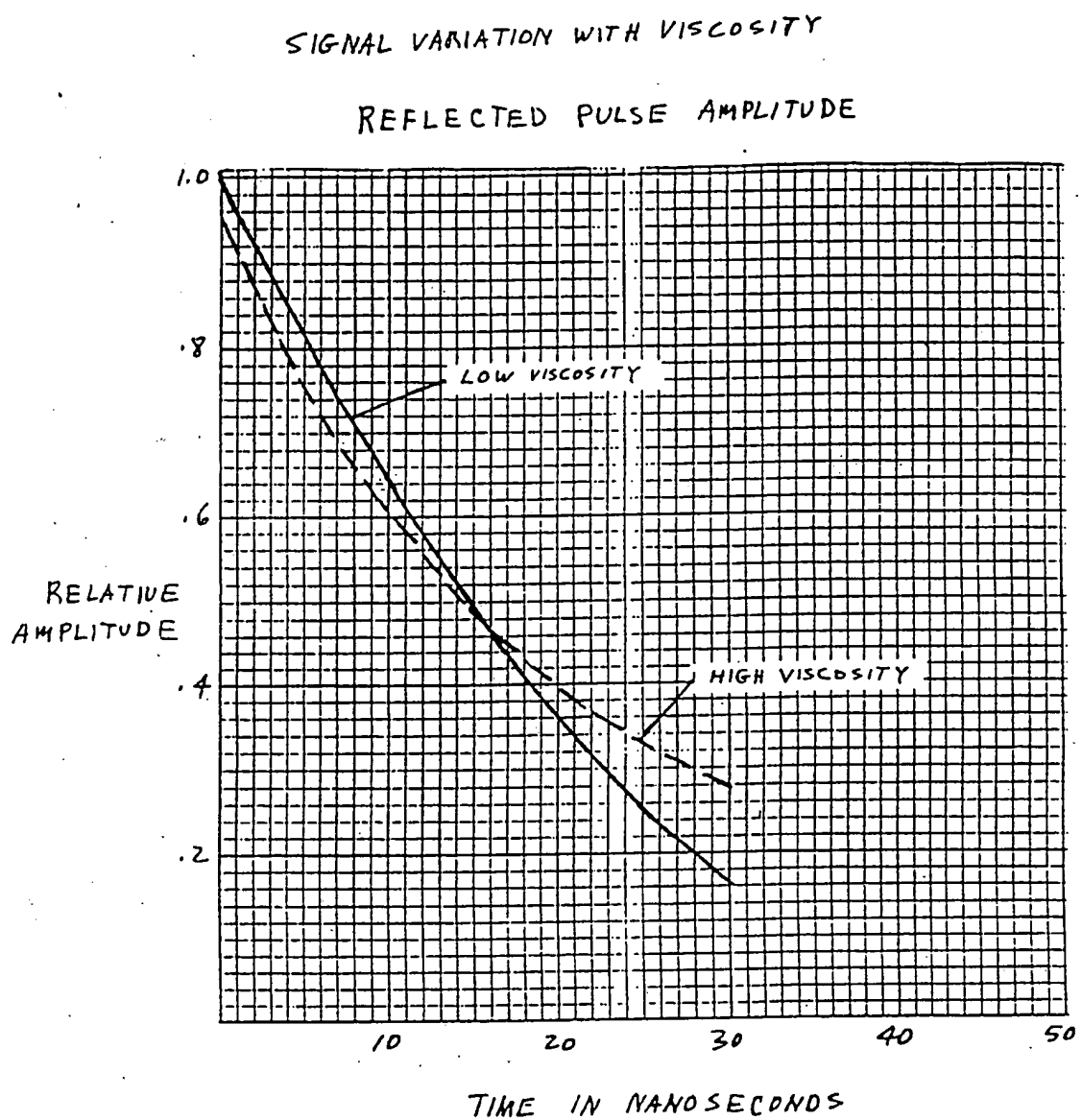


FIG. 5

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SIGNAL VARIATION WITH DIPOLE ROTATION  
REFLECTED PULSE AMPLITUDE

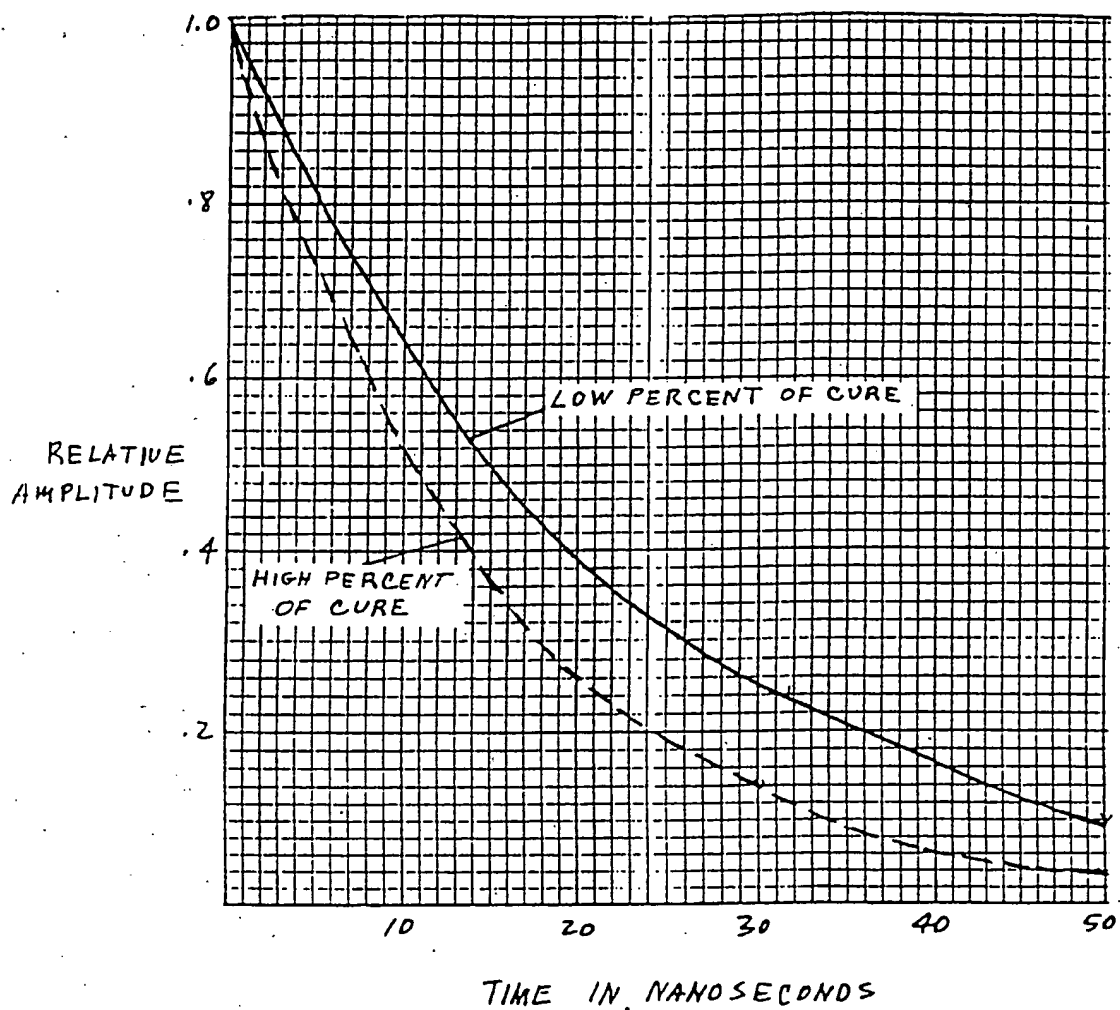


FIG. 6

## INTERNATIONAL SEARCH REPORT

 International application No.  
PCT/US98/18736

## A. CLASSIFICATION OF SUBJECT MATTER

 IPC(6) :G01R 27/04  
US CL :324/637, 642, 71.1

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 324/632, 637, 639, 640, 642, 643, 646, 71.1; 73/54.41

 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
none

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4,779,452 A (COHEN-TENOUDJI et al) 25 October 1988, (25/10/88), see Fig. 5.	1-13
A	US 5,334,941 A (KING) 02 August 1994, (02/08/94), see Figs. 1-8.	1-13
A,P	US 5,744,971 A (CHAN et al) 28 April 1998, (28/04/98), see Figs. 1 and 2.	1-13
A,P	US 5,748,002 A (SCOTT et al) 05 May 1998, (05/05/98), see Figs. 1-6.	1-13

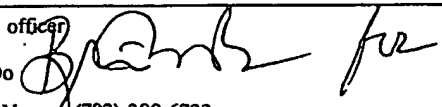
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